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(21) International Application Number: PCT/HU96/00008 (22) International Filing Date: 16 February 1996 (16.02.96) (71) Applicant (for all designated States except US): FAIPARI KUTATÓ INTÉZET KFT. [HU/HU]; Vörösmarty út 56, H-1201 Budapest (HU). (72) Inventors; and (75) Inventors/Applicants (for US only): HADNAGY, József [HU/HU]; Szász K. út 2-4, H-1027 Budapest (HU). JUHÁSZ, A., Zoltán [HU/HU]; Szőlő köz 5, H-1032 Budapest (HU). KIRÁLY, Béla [HU/HU]; Andor út 14, H-1119 Budapest (HU). SZABÓ, Péter [HU/HU]; Viola út 20, H-8200 Veszprém (HU). (74) Agent: DANUBIA; P.O. Box 198, H-1368 Budapest (HU).		(81) Designated States: AL, AM, AT, AU, AZ, BB, BG, BR, BY, CA, CH, CN, CZ, DE, DK, EE, ES, FI, GB, GE, HU, IS, JP, KE, KG, KP, KR, KZ, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, TJ, TM, TR, TT, UA, UG, US, UZ, VN, ARIPO patent (KE, LS, MW, SD, SZ, UG), Eurasian patent (AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, ML, MR, NE, SN, TD, TG). Published <i>With international search report.</i>
(54) Title: PROCESS FOR THE PRODUCTION OF GYPSUM-BASED COMPOSITE BOARDS AND SHAPED BODIES (57) Abstract The invention relates to a process for producing gypsum-based boards and shaped bodies from a material containing calcium-sulfate dihydrate ($\text{CaSO}_4 \times 2\text{H}_2\text{O}$) mixed with skeleton material and fillers. In the process a gypsum-matrix formation, respectively hardening, based on the recrystallization of dihydrate, take place instead of the commonly known de- and rehydration processes. The state of change is achieved by working with hot water above the saturation vapour pressure, preferably on the pressure/temperature function curve of dihydrate or under a higher pressure. In the board manufacturing process according to the invention, the boards are pressed at a temperature of 110 - 200 °C, preferably 130 - 180 °C under a pressure of 0.2 - 5.5 MPa, preferably 0.4 - 2.5 MPa. A mixture of 1 part by weight of hemihydrate flour, 0.05 - 0.35 part by weight of fillers and skeleton material (mostly waste paper fibers), 0.05 - 0.3 part by weight of water and preferably 0 - 0.05 part by weight of a sulfate-containing substance is used as base material mixture. In the shaped body manufacturing process according to the invention, the preheated suspension is introduced to a closed mould at a temperature of 110 - 200 °C, preferably 130 - 180 °C, under a pressure of 0.2 - 5.5 MPa, preferably 0.4 - 2.5 MPa. Under the overpressure established there, the mixture hardens while cooling down. A mixture of 1 part by weight of dihydrate flour, 0.3 - 1.5 part by weight of water, 0 - 0.3 part by weight of a filler and an additive preferably containing 0 - 0.05 part by weight of sulfate is used as base material mixture.		

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PROCESS FOR THE PRODUCTION OF GYPSUM-BASED COMPOSITE BOARDS AND SHAPED BODIES

5 The invention relates to a process for the production of composite boards and shaped bodies, totally consisting of gypsum or gypsum mixed with skeleton materials and fillers. Instead of the commonly known calcium sulfate hemihydrate ($\text{CaSO}_4 \times 1/2\text{H}_2\text{O}$; hereinafter hemihydrate), calcium sulfate dihydrate ($\text{CaSO}_4 \times 2\text{H}_2\text{O}$; hereinafter dihydrate) is used as base material in the process according to the invention.

10 For the production of gypsum-based composite boards essentially methods of two kinds are known [P. Takács: Faipar (in English: "Woodworking Industry") 8, 140-149 (1993)]. According to one of these, hemihydrate being the base material of the process is produced from dihydrate in a separate gypsum-calcinating furnace. This binding material is mixed with water required for the
15 hardening of hemihydrate, fillers and skeleton materials as well as other materials. A mat is formed from the mixture, which is then pressed. During compression the gypsum hardens and the board is formed out. The moisture content of the product is adjusted by an artificial final drying. The process is called as wet, half-dry or dry process, depending on the amount of water used
20 for mixing-in as well as the technological site and time of water addition.

According to the other method, a dihydrate flour is utilized as base material. A suspension is prepared from dihydrate, fillers and skeleton materials, other additives as well as water. Therefrom, a mat is first prepared, which is then dehydrated. Dihydrate forming the main constituent of the mat is first calcinated
25 (dehydrated) in a heat-press or autoclave, then the hemihydrate obtained is rehydrated with water at the same place, simultaneously with the decrease in temperature and pressure conditions. The moisture content of the hardened board is adjusted similarly by a final drying.

There exists no difference in the basic principle of these traditional techniques. Each of them is based on the dehydration and rehydration of gypsum known for several hundreds of years. The basic principle is not altered by carrying out the procedures of crystal water loss and re-incorporation (de- and rehydration) in a single step or several steps, respectively.

Thus, as it is known e.g. from the German published patent application No. 34 19558 A1, the processes of dehydration and rehydration are carried out in the same autoclave. In this process, an aqueous suspension is prepared from a mixture containing finely ground dihydrate, a fibrous material (e.g. waste paper fibres) in an amount of not more than 10 % by weight as well as substances controlling the hardening. After being applied onto an endless screen belt, the dilute slurry-like suspension is dehydrated to a moisture content of less than 40 % by weight in a belt press by the simultaneous use of vacuum and pressure. After cutting the endless mat to the desired size, the raw boards are first calcinated in an autoclave, then rehydrated, in a single run. The temperature of the autoclave is adjusted first to 135-145 °C, then below 100 °C to transform the hemihydrate formed again to dihydrate. The temperature of 142 °C and pressure of 4 bar defined in an example of the description is one point of the saturated vapour curve.

Other known processes may be considered to be the further developed variants of the former basic conception. Thus, e.g. according to the European published patent application No. 0 173 351 A2, a difference consists only in that the hemihydrate is prepared in a heat-press but not in an autoclave. It appears from the European published patent application No. 0 309 709 A1 that in the first step the boards are calcinated at 100-170 °C under atmospheric pressure in an autoclave, then they are hardened, i.e. rehydrated, with water at the same place. The process according to European published patent application No. 0 294 566 A1 is a further example of diminishing the drawbacks of former processes; according to this water containing also hardening-accelerators is let flow through the calcinated boards.

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As the most important disadvantages of the known processes, it can be noted that a significant energy loss occurs because of the repeated heating of equipments of large weight; furthermore, the de- and rehydration processes may last for several hours.

- 5 Thus, the present invention is aimed to produce from dihydrate gypsum-comprising composite boards and shaped bodies with favourable technological parameters by eliminating the drawbacks of former processes, beside lower investment and manufacturing costs.

10 The invention is based on the recognition that during the transformation taking place under pressure/temperature defined hereinafter, the dihydrate being the base material, consisting of separate crystals, recrystallizes [the crystals "join in growing" (coalesce), their size increases, their form is changed] and forms a gypsum-matrix [which is the base of formation of any gypsum-based artificial stone] without undergoing any de- and rehydration process.

- 15 Thus, according to our recognition, the condition of transformation lies therein that the pressure of the medium surrounding the dihydrate should be higher than the partial vapour pressure of dihydrate. On comparison to the traditional processes, the significant difference consists in that the traditional processes are carried out according to the pressure/temperature function of the saturation vapour pressure or under a lower pressure; whereas, according to the present invention the process is carried out above the saturation vapour pressure, preferably according to the pressure/temperature function of the dihydrate or even under higher pressures. The term "pressure" is meant to be the pressure inside the board or shaped body. This condition has not been realized in any of the former processes; therefore, the recrystallization according to the invention cannot take place therein even temporarily. The summarizing handbook entitled "Gmelins Handbuch der Anorganischen Chemie" [Vol. 8, No. 28, Part 1; Verlag Chemie/Weinheim (1961)] contains nearly all basic data relating to the chemistry of gypsum. It appears from this handbook that a number of researchers [e.g. Van't Hoff (1900) as well as Toriumi (1938)] investigated the
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determination of the constitutional diagram of gypsum. The basic data occurring here are being used in a wide range at present, too. All researchers studied only the state changes of dihydrate occurring at a pressure below the pressure/temperature function curve; the part above this function curve was
5 not investigated because the dihydrate was considered to be in equilibrium state in this region.

Thus, the invention relates to a process for the production of gypsum-based composite boards and shaped bodies from a mixture of calcium sulfate dihydrate as skeleton material, water and usual additives. The process
10 according to the invention comprises hardening the mixture by water having a pressure exceeding the saturation vapour pressure.

A practical embodiment of process according to the invention is accomplished as follows.

In order to achieve the appropriate crystal forms, compounds containing
15 sulfates, e.g. aluminum sulfate, may be used as additives which, in addition to shaping out a more favourable crystal structure of gypsum, accelerate the recrystallization process, too.

The native or artificial fillers and skeleton materials designed to the reinforcement of products prepared by using the process of the invention may
20 be either of organic origin (preferably chips, cellulose fibres, synthetic filaments) and/or inorganic origin (preferably glass fibres, carbon fibres). Fillers and skeleton materials are meant to be fibrous or granular materials endowing the products with more favourable parameters, as well as a group of materials, by the mixing-in of which the material costs can be diminished. The
25 transformation process is not bound to the condition of presence of fillers and skeleton materials or additives; in addition, these are known materials [G.Kühne: Baustoffindustrie 2, 40-46 (1989)].

In the process according to the invention, the pressure, being the function of the temperature, has no theoretical but technical limits. By elevating the

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temperature by 20-30 °C the time of treatment can be decreased to a fraction thereof; however, due to the necessity of a nearly exponential increase in the pressure, robust equipments with too large weight would be to be employed at high temperatures. Thus, it is unsuitable to increase the temperature above 180 °C.

According to the demands of utilization, the parameters of products produced by using the process according to the invention can be varied within a wide range. Thus, e.g. both a light product with a density of 900 kg/m³ and a bending strength of 5 MPa as well as a product with a density of 1400 kg/m³ with a strength of 3 times as high as that of the former one, may be produced.

One of the fields of preferred utilizations of the invention is represented by the techniques of the gypsum-based board manufacturing. This can be achieved on the plant-like working production line designed to the manufacturing of carbondioxide-hardened cement boards (M.Tompa: UNITED NATIONS TIM/SEM, 1991/14 R5). The base material of fillers and skeleton materials should be chopped, torn to the fibres thereof in the traditional manner. Paper waste is the most frequently used one of such materials. Optionally, the ground dihydrate is mixed together in an agitator with the optional additives, obligatorily with skeleton materials and fillers as well as with a small amount of water needed to adhesion and plastification. A mat is formed from the mixture. The principles of mat formation do not differ from the usual ones. The mat formation is carried out on a sectioned screen. The mat together with the screen gets to the unheated, one-level hydraulic press of large board size, closing along the edges, too. The plates of press are provided with bores perpendicular to the plate plane for introducing and leading out hot water. After closing the press, the board is pressurized with hot water through these bores. The gypsum-matrix is formed out, the board hardens within a short period. Subsequently, the water is emptied through the bores mentioned, then the press is opened. The hot boards coming from the press are transferred to the final dryer. After this, edging and packaging only remain. There is no additional

hardening. The hot water flowing out is utilized as mixing water for the next boards; thus, no waste water is formed. During the process, gypsum is present in its dihydrate state in any moment. The system of the press and other technical equipments and mode of heat-transfer may be varied depending on the demand.

An other significant field of utilizing the process according to the invention is represented by the production of gypsum-based building blocks and shaped bodies. Traditionally, the blocks are produced by mixing the calcinated gypsum (hemihydrate) with water and pouring the slurry into a mould, where it hardens and can be removed from the mould. Subsequently, the excess moisture is removed by drying. Any process for manufacturing gypsum blocks by the direct utilization of dihydrate as base material was till now unknown.

An other embodiment of the process according to the invention comprises forming a suspension from ground dihydrate, water and granular additives and heating them above 100 °C in an autoclave. The hot mixture is carried into a closed mould by further enhancement of the pressure. Due to the partial pressure above the equilibrium pressure of dihydrate, the substance recrystallizes, hardens, i.e. forms a gypsum-matrix while cooling. The shaped bodies cooled down are removed from the mould and the excess moisture is removed by drying.

The process according to the invention provides the following technical and economical advantages in comparison to the wet, half-dry and dry processes of board manufacturing.

- It is unnecessary to calcine gypsum representing 70-80 % of the weight of gypsum fibre boards but ground gypsum stone can be used without any pretreatment.
- Not only a ground gypsum stone flour but also chemical and waste-gas gypsum (REA) as well as gypsum moulds of ceramic industry unuseful for re-utilization are suitable for using in the process of the invention. Waste-gas

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gypsum is most useful to this purpose because, due to its perfect particle structure, it can be used without any pretreatment (briquetting, drying, calcinating, grinding). The waste-gas gypsum forms have a higher dihydrate content and a board of higher strength can be produced therefrom.

- 5 - Lower power expenditures are necessary for grinding the gypsum-stone (in the cases of gypsum moulds and mineral gypsums), which is accompanied by less technical problems.
- 10 - The coarse and ground gypsum-stone, waste-gas gypsum can be stored even at an uncovered place. The rain precipitation and freezing do not induce any alteration therein, nor induce any hardening process as e.g. in the case of calcined gypsum or cement. The storability of dihydrate is unrestricted.
- 15 - A mixture prepared with strengthening materials and/or fillers as well as additive water can also be stored for an unrestricted period in opposition to the wet technological processes, wherein the hardening begins at the moment of adding water and cannot be stopped. In the case of breakdown, particularly in the techniques using belt press or clamping device systems, the mixture hardens in the agitator [P. Takáts: Faipar 8, 140-149 (1993)]. In the process according to the invention the hardening begins only in the
20 press.
- 25 - The total amount of waste material from edging and faulty rejected boards can be recycled; therefore, no waste is formed. The techniques are fully unharmed to the environment.
- Normally, extracted substances dissolved from the wood and other aggregates disturb the hardening of gypsum or cement. In the case of the process of the invention this does not mean any problem since they cannot exert a harmful effect in this medium and even promote the development of a favourable crystal structure.

- The total investment of the gypsum-calcinating furnace can be saved, which represents about 30-40 % of the total investment costs (calcinating furnace and board production line) for a complex technological process.
- 5 - The total heat demand (about 1200 MJ/ton) and electric power demand (about 30 kW/ton of hemihydrate) of the calcinating furnace are left out. During heat-pressing, the heat used to warm up the boards and to cover heat losses are required. During transformation, the heat used to warm up the boards are only to be invested since the transformation per se is neutral. Only a small fraction of the heat allocated to calcination of gypsum is
10 required for the heat-pressing.
- A smaller plant building is required. The techniques according to the invention demand less space than the traditional ones since the space demand of the equipments is less and the calcinating furnace is unnecessary. The major part of the techniques can be composed of
15 previously developed and widely used equipments.
- Smaller dryers and less heat are required because the board leaving the press gets as hot to the dryer; therefore, the heat required to warm up the board can be saved.

20 On comparison to the dihydrate-based board manufacturing processes, the process according to the invention possesses further advantages as follows.

- In the former processes the time of re- and dehydration may last several hours; this is decreased to a few (2-10) minutes in the process based on the principle of recrystallization.
- 25 - Due to the repeated heatings and coolings of large-weight equipments, stresses can develop in the equipments and much energy gets lost in the course of these procedures. In the process according to the invention the cooling-back of the equipments is unnecessary.

- Due to the long-lasting treatment, substances inhibiting the hardening process are dissolved from the skeleton material (e.g. waste paper). This problem does not occur in the process according to the invention.

5 The process according to the invention is illustrated in more detail by the following non-limiting Examples.

Example 1 (The techniques of board manufacturing)

A homogeneous mixture is formed from 0.2 part by weight of waste paper chopped to fibres, 1 part by weight of dihydrate flour of sieve fraction below 0.2 mm, 0.2 part by weight of water and 0.001 part by weight of aluminum sulfate.
10 From this mixture, a mat of nearly 5 cm thickness is formed on a sectioned screen. The mat enters the press together with the screen. After closing the press, water with 1.4 MPa pressure and 160 °C temperature is pressed into the board through the bores perpendicular to the board plane for 5 minutes. At the
15 end of the pressing period, the inner pressure of board is diminished down to the atmospheric pressure through the above bores and the press is opened. Then, the ready boards are dried in a board-dryer to a moisture content of nearly 3 % by weight. Parameters of the final product are: a bending strength of 8 MPa and density of 1150 kg/m³.

On comparison of the process described in above Example 1 to the process
20 known from the German published patent application No. 34 19558 A1, a product with nearly the same parameters is prepared from a mixture of materials with nearly the same composition by input of energy being less by an order of magnitude. This is due to the fact that, in the process of invention, no equipment of large weight is heated to a working temperature and cooled down
25 below 100 °C in each working cycle. The parameters are also significantly different. On operating both equipments e.g. at 160 °C, saturated vapour with 0.63 MPa pressure is introduced to the autoclave for nearly 2 hours in the process used for comparison; whereas hot water with 1.4 MPa pressure is used for 5 minutes in the process according to the invention.

Example 2 (Techniques of the shaped body manufacturing)

5 A mixture is prepared from 1 part by weight of dihydrate flour, 0.6 part by weight of water and 0.01 part by weight of aluminum sulfate and heated up to 170 °C in an autoclave. The hot mixture is filled into a closable mould and a pressure of 1.7 MPa is established there by diminishing the inner volume of mould. While cooling down within about 10 minutes, the product hardens. The mould can be opened at 100 °C and the shaped body can be removed. Then, the shaped body is air-dried. Parameter of the product is a density of 900 kg/m³.

C L A I M S

1. Process for the production of gypsum-based composite boards and shapes from a mixture of calcium sulfate dihydrate, skeleton material, water and optionally other additive material(s), which c o m p r i s e s hardening the mixture by using water having a pressure exceeding the saturation vapour pressure.
2. A process as claimed in claim 1, which c o m p r i s e s carrying out the hardening of the mixture in a temperature range between 110 °C and 200 °C, preferably between 130 °C and 180 °C.
3. A process as claimed in any of the claims 1 to 3, which c o m p r i s e s using a calcium sulfate dihydrate sieve fraction below 0.2 mm, preferably below 0.05 mm.
4. A process as claimed in any of the claims 1 to 3, which c o m p r i s e s using as a source of calcium sulfate dihydrate a mineral flour and/or chemical gypsum wastes, preferably waste-gas gypsum and/or phosphogypsum as well as gypsum mould flour of ceramic industry unuseful for re-utilization.
5. A process as claimed in claim 1 for the production of a composite board, which c o m p r i s e s bringing water with a pressure exceeding the saturation vapour pressure and a solution containing base materials in between the press plates.
6. A process as claimed in any of claims 1 to 4 for the production of a shaped body, which c o m p r i s e s cooling down the preheated suspension in a closed mould after hardening.
7. A process as claimed in claim 6, which c o m p r i s e s carrying out the cooling down within a period shorter than one hour, preferably during 2 to 10 minutes.

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8. A process as claimed in any of the claims 1 to 7, which c o m p r i s e s adjusting the weight ratio of skeleton material and fillers to dihydrate to a value of 0-0.35, preferably 0.1-0.25.

INTERNATIONAL SEARCH REPORT

International Application No

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C04B40/02 C04B28/14

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 6 C04B

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US,A,1 652 162 (BROOKBY) 13 December 1927 see page 1, line 45 - line 74 see claims ---	1,4
A	US,A,4 443 261 (NORDQVIST) 17 April 1984 see the whole document ---	1,4
A	EP,A,0 166 094 (BABCOCK-BSH AKTIENGESELLSCHAFT) 2 January 1986 cited in the application see the whole document ---	1,2,4
A	GB,A,2 066 235 (PENTTI HUKKANEN) 8 July 1981 see page 2, line 4 - line 48 see claims ---	1,4
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C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT		
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